इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 12628 (1989): Dye Intermediates - 2-Amino-4-Nitrophenol, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN
IGNORANTIA JURIS NON EXCUSAT
PROTECTED BY COPYRIGHT

(Reaffirmed 2000)

Indian Standard

DYE INTERMEDIATES — 2-AMINO-4-NITROPHENOL, TECHNICAL — SPECIFICATION

भारतीय मानक

2-एमिनो-4-नाइट्रोफिनाल तकनीकी – विशिष्टि

UDC 667·289 : 547·564·3

© BIS 1989

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1989

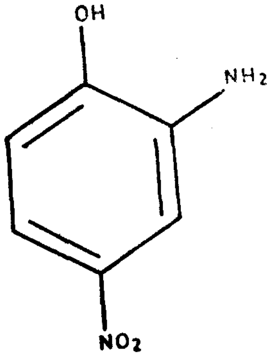
Price Group 2

Dye Intermediates Sectional Committee, PCDC 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 27 January 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2-amino-4-nitrophenol ($C_6H_6O_3N_2$) is used as an intermediates for reactive dyes. It is represented by the following structural formula:



2-Amino-4-Nitrophenol (Molecular Mass 154) CAS Registry Number [99-57-0]

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DYE INTERMEDIATES — 2-AMINO-4-NITROPHENOL, TECHNICAL – SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2-amino-4-nitrophenol, technical.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard.

IS No.	*Title*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision*).
IS 2552 : 1979	Specification for steel drums (galvanized and ungalvanized) (*second revision*)
IS 5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be dry or moist, blackish brown to black in colour.

3.2 The material shall also comply with requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1979) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer, and his recognized trade-mark, if any;

c) Batch number; and

d) Gross, net and tare mass.

Table 1 Requirements for 2-Amino-4-Nitrophenol, Technical

(*Clauses* 3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Matter insoluble in dilute hydrochloric acid, percent by mass, *Max*	2·0	A-2
ii)	Assay (on dry basis), percent by mass, *Min*	60·0	A-3
iii)	Colour of diazotized filtered solution	To pass the test	A-4
iv)	2-aminophenol, percent by mass, (on 100 percent basis), *Max*	0·50	A-5
v)	2 : 4-dinitrophenol, percent by mass (on 100 percent basis) , *Max*	0·50	A-5

5 SAMPLING

5.1 The material shall be sampled in accordance with 3 of IS 5299 : 1969.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annex A, as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1)

METHODS OF TEST FOR 2-AMINO-4-NITROPHENOL, TECHNICAL

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 DETERMINATION OF MATTER INSOLUBLE IN DILUTE HYDROCHLORIC ACID

A-2.1 Reagents

A-2.1.1 *Hydrochloric Acid,* 30 percent.

A-2.1.2 *Congo Red Paper*

A-2.2 Apparatus

A-2.2.1 *Beaker,* 1000-ml.

A-2.2.2 *Sintered Glass Crucible,* G 3.

A-2.3 Procedure

Weigh accurately about 15 g of *prepared sample* (**A-1.1**) and transfer it to a 1 000-ml beaker. Add 300 ml hot water and hydrochloric acid till acidic to Congo Red Paper. Heat to dissolve completely. Filter hot through sintered glass crucible and wash the residue with hot water till free from acid. Preserve the filtrate and washings for assay as given in **A-3** and dry the residue at 100°C to a constant mass.

A-2.4 Calculation

$$\text{Matter insoluble in hydrochloric acid, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of sintered crucible with residue,

M_2 = mass in g of empty sintered crucible, and

M = mass in g of sample taken for test.

A-3 ASSAY

A-3.1 Reagents

A-3.1.1 *Concentrated Hydrochloric Acid*

A-3.1.2 *Sodium Nitrite Solution* ($NaNO_2$) , N/10.

A-3.1.3 *Starch Iodide Paper*

A-3.2 Procedure

Cool the filtrate (**A-2.3**) and washings to room temperature, dilute with distilled water to 1 000 ml in a volumetric flask and mix well. Pipette out 100 ml aliquot in a 1 000-ml beaker. Add 300 ml ice cold water and 50 ml of concentrated hydrochloric acid. Cool to 10°C by adding ice and titrate while stirring mechanically at 10°C, with standard N/10 sodium nitrite, using potassium starchiodide paper to a 5 minute end point.

A-3.3 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 154}{M}$$

where

V = volume of N/10 $NaNO_2$ required in ml,

N = normality of standard $NaNO_2$, and

M = mass in g of sample taken for test.

A-4 DETERMINATION OF COLOUR OF DIAZOTIZED FILTERED SOLUTION

A-4.1 Reagents

A-4.1.1 *Concentrated Hydrochloric Acid*

A-4.1.2 *Sodium Nitrte Solution,* 2 N

A-4.1.3 *Starch Iodide Paper*

A-4.2 Apparatus

A-4.2.1 *Buchner Funnel*

A-4.2.2 *Ice-Bath*

A-4.3 Procedure

Weigh about 20 g of *prepared sample* (**A-1 1**) and transfer it to a 250-ml beaker. Add 100 ml distilled water and 10 ml of concentrated hydrochloric acid. Stir for 30 minutes at room temperature and filter through Buchner funnel using Whatman No. 1 filter paper. Do not wash. Transfer the filtered solution in a 500-ml beaker and cool to 5°C externally in a ice-bath. While stirring mechanically, add sodium nitrite solution till excess nitrite gives a positive test on potassium starch iodide paper for 10 minutes, without further addition of nitrite.

A-4.3.1 The sample shall be taken to have passed the test if the colour of the diazo slurry is not darker than pale brown.

A-5 DETERMINATION OF 2-AMINOPHENOL AND 2 : 4-DINITROPHENOL

A-5.1 Outline of the Method

The impurities in 2-amino-4-nitrophenol are estimated by thin layer chromatography.

A-5.2 Apparatus

A-5.2.1 *Chromatographic Chamber,* 22 × 10 × 22 cm.

A-5.2.2 *Micropipette,* 10 μl capacity.

A-5.2.3 *Thin Layer Chromatographic Glass Plates,* 20 × 20 cm.

A-5.2 4 *Adjustable Thin Plate Applicator*

A-5.2.5 *Chromatographic Spray Bottle*

A-5.2 6 *Oven,* with a thermo regulator.

A-5 2.7 *Weighing Bottle*

A-5.3 Reagents

A-5.3.1 *Solution of 2-Aminophenol in Methanol,* 0·01 percent (*m/v*) (reference solution).

A-5.3.2 *Solution of 2:4-Dinitrophenol in Methanol,* 0·01 percent (*m/v*) (reference solution).

A-5.3.3 *Eluent,* chloroform : methanol (98:2) in acetic acid.

A-5.3.4 *Coupling Reagents,* 1 percent of 4-dimethylaminobenzaldehyde and 10 percent solution of stannous chloride (1:1).

A-5.3.5 *Silica Gel-G*

A-5.4 Procedure

A-5.4.1 *Preparation of the Cromatographic Plates*

Mix 50 g of neutral silica gel with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding 0·25 mm and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 30 mm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-5.4.2 *Preparation of the Sample Solution*

Homogenize the prepared sample for a few minutes and weigh by means of a weighing bottle 2·0 g of the material and transfer to a 100-ml volumetric flask. Dissolve in acetone and make up the solution to the mark by adding distilled water.

A-5.4.3 Spot 10 μl of the sample solution (**A-5.4.2**) and those of reference solutions (**A-5.3.1** and **A-5.3 2**) on the thin layer plate. Place the eluent in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the eluent to run in ascending manner for about 100 minutes. Remove the plate from the chamber and dry off the solvent in air drier. Spray the plate with coupling reagent (**A-5.3.4**).

A-5.4.3.1 By visual comparison, the spots corresponding to 2-aminophenol and 2:4-dinitrophenol in the sample shall have equal or less intensity than that of the reference solutions.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.